United States Patent Application

Title:

Nitric Oxide purification method and apparatus.

Luca D'Ottone **March, 1st, 2004**

Description

BACKGROUND OF THE INVENTION

[0001] 1. Field of the Invention

[0002] The present invention relates to a novel system for the purification of nitric oxide. The present invention also relates to a method of producing nitric oxide of ultra-high purity. The invention was developed to provide impurities free nitrogen monoxide for research purposes in

a limited budged research laboratory. The process was found to be extremely effective and has potential for scale up.

[0003] 2. Description of the Related Art

[0004] Nitric oxide is a colorless gas having extremely high reactivity toward atmospheric oxygen and other oxidant present in the atmosphere like the hydroxyl radical. NO reacts quickly with these oxidants becoming NO_2. NO finds a number of critical applications both in the pharmaceutical and in the semiconductor industry.

[0005] It is particularly important for nitric oxide to be of ultra high purity, on the order of 99.999% or greater purity. Such purity will ensure the absence of dangerous NO_2 in medical applications and limit the presence of other impurities like SO_2, SO_3, and CO_2 in nitrogen monoxide for industrial uses.

[0006] A number of US patents deal with methods to purify nitric oxide: 5,670,125, 5,514,204, 5,417,950, 5,268,465. All of these methods use a zeolite filter to free nitrogen monoxide from impurities.

[0007] The main disadvantages of all these methods is that a zeolite filter is not something commercially or readily available in research labs, it is expensive, and must be changed due to chemical deterioration.

[0008] Another patented method to purify nitric oxide is US patent 6,576,044, but it also suffer of the main disadvantage to require extensive equipment and/or specific chemicals.

[0009] The method, object of this application, allows the purification of nitric oxide for industrial and research purposes at little or moderate cost. It's easy to use and set up, and does not require expensive parts. It is particularly indicated for those applications where it is critical to remove acidic impurities like NO_2, SO_2, SO_3, and CO_2 from the gaseous mixture.

[0010] A further advantage associated with the novel system is that the total cost of purification can be significantly reduced by eliminating complicated secondary operations needed to eliminate carbon dioxide and light impurities

like volatile organics.

[0011] It is a specific object of the invention to provide a process for the removal of acid gases from nitric oxide. In the past there have been some attempt to achieve this level of purity for research purposes but results using literature methods are not satisfactory. [Hughes, 1961]

[0012] A similar apparatus have been suggested to purify nitrous oxide (not nitric oxide) in the US published application 20020056289. No reference is made in such application to the use of that apparatus for purifying nitric oxide.

[0013] Still another difference from US published application 20020056289 is that we found effective the use of a mixture of hydroxides of alkali and earth alkali metals for the first filter, instead of the use of ASCARITE(registered trademark) only.

[0014] In addition to that it is a goal of this invention to eliminate N_2O as a potential impurity contained in the initial mixture. This invention achieves that by freezing out the N_2O in the first filter. In one of the possible

embodiments of this invention the first filter is kept at the temperature around 150 K. At this temperature the nitrous oxide, having freezing point at 182.32 K, freezes on the wall of said first filter, while the nitric oxide, having freezing point at 109.54 K does not solidify and can flow to the second filter without being depleted. One optional feature of this invention is a specially designed filter net kept at a constant temperature to freeze out nitrous oxide and all other impurities having a condensation point higher than the one of nitric oxide.

[0015] Other objects and aspects of the present invention will become apparent to one of ordinary skill in the art on a review of the specification, and claims appended thereto.

SUMMARY OF THE INVENTION

[0016] This invention consists in an innovative process and a novel apparatus for purification of nitric oxide. The invention finds particular applicability in the semiconductor manufacturing industry, in research applications, and in the medical industry.

[0017] The process for removal of impurities from a nitric oxide includes the steps of:

[0018] (a) providing a mixture of impure nitric oxide and, optionally, an inert gas in a purification tank;

[0019] (b) routing the impure nitric oxide trough a filter composed by a mixture of hydroxides of alkali and earth alkali metals kept at around 150 K.

[0020] (c) collecting the purified nitric oxide in a delivery tank.

[0021] (d) preventing the entrance of oxygen or other contaminants in the delivery tank.

[0022] In accordance with another aspect of the invention an apparatus for nitric oxide purification is provided. The system includes: (a) a tubing or plumbing system having its internal surface covered with an inert material; (b) two filter chambers having its internal surface covered with an inert material; (c) a first filter consisting of a mixture of hydroxides of alkali and earth alkali metals; (d) a second filter consisting of a molecular sieve; (e) a thermostatic chamber to keep constant the temperature of the filter chamber; (f) a temperature sensor to monitor the temperature of the filter chamber.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS OF THE INVENTION (i.e. DESCRIPTION OF THE DRAWINGS)

[0023] The invention provides an efficient and effective means of purifying a nitric oxide from common impurities like acid gases. The term acid gases as used herein is defined as carbon dioxide, nitrogen dioxide, sulfur dioxide, sulfur trioxide, and other acid gases known to the artisan skilled in the art.

[0024] The invention provides an efficient method to purify nitric oxide from compounds having a freezing point higher than 109.54 K.

[0025] The apparatus in claim fourteen can be described with reference to the figure (1) of drawing.

[0026] Figure (1) Number (1) in the drawing: the delivery tank.

[0027] Figure (1) Number (2) in the drawing: first tubing system internally coated with inert material.

[0028] Figure (1) Number (3) in the drawing: a needle valve to regulate the gas flow trough the filter system.

[0029] Figure (1) Number (4) in the drawing: thermostatic jacket around the first filter.

[0030] Figure (1) Number (5) in the drawing: first filter: a filter chamber internally coated by inert material such as Teflon (registered trade mark), filled with a mixture of hydroxides of alkali and earth alkali metals.

[0031] Figure (1) Number (6) in the drawing: second tubing system internally coated with an inert material.

[0032] Figure (1) Number (7) in the drawing: thermostatic bath containing the refrigerant.

[0033] Figure (1) Number (8) in the drawing: second filter: a filter chamber internally coated with an inert material filled with a molecular sieve.

[0034] Figure (1) Number (9) in the drawing: a collection tank.

[0035] Figure (1) Number (10) in the drawing: a vacuum pump.

[0036] Figure (2) illustrates said first filter system, composed by a metal filter-net, a box internally covered by an inert material, an internal filter composed by a mixture of hydroxides of alkali and earth alkali metals, a support of silica dioxide.

[0037] Figure (2) Number (1) in the drawing: metal filter-net.

[0038] Figure (2) Number (2) in the drawing: connections to an external cooling system for the metal filter net.

[0039] Figure (2) Number (3) in the drawing: internal filter system composed by a support of silica dioxide and having as an active component at least one hydroxide of alkali or earth alkali metals.

[0040] Figure (2) Number (4) in the drawing: a box internally covered by an inert material such as Teflon (registered Trade mark), containing the filter material.

[0041] Figure (2) Number (5) in the drawing: connections to an external cooling system for the filter box.

EXAMPLE

[0042] A nitric oxide purification system as shown in the drawing was used to purify a mixture of nitric oxide in Helium. The mixture of impure nitric oxide (10%) in Helium (balance) is flushed from a delivery tank at above atmospheric pressure trough the first filter.

[0043] The first filter has two functions.

[0044] The first function of the first filter is to remove all the impurities from the flow of nitric oxide having freezing point above the freezing point of nitric oxide i.e. higher than 109.54 K. This is achieved by keeping the temperature of the filter as close as possible to 110 K. Such impurities include NH_3, N_2O, H_2O, hydrocarbons.

[0045] The second function is to eliminate all the acid impurities by neutralization on the filter composed by a mixture of hydroxides of alkali and earth alkali metals. Such impurities include CO_2, SO_2, SO_3, HNO_3, NO_2.

[0046] The first filter, once exhausted, cannot be regenerated and must be changed.

[0047] The nitric oxide is then passed through a second filter. The second filter is constituted by a commercial molecular sieve.

[0048] The nitric oxide is then collected in a collection tank.

[0049] The purity of the nitric oxide is checked by UV spectroscopy using an optical multi-channel analyzer (OMA). Nitric oxide has negligible cross section in the UV visible region of the electromagnetic spectrum.

[0050] The concentration of nitric oxide in the collection tank is estimated by titrating the nitric oxide with oxygen and comparing the recorded spectra with the absolute cross section of nitric dioxide.

[0051] The presence of impurities can also be monitored by a gas chromatography (GC) model VARIAN 3400 capable of detecting to as low as 0.01 part per million by volume (ppmv).

[0052] The invention can also function without the second filter, that is in fact optional.

[0053] While the invention has been described in detail with reference to a specific embodiment thereof, it will be apparent to one skilled in the art that various changes and modifications can be made, and equivalents employed without departing from the scope of the claims.